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PROVISIONAL SPECIFICATION

Applicant(s):

SHI XUE DOU

Invention Title:

SUPERCONDUCTING MATERIAL AND METHOD OF SYNTHESIS

The invention is described in the following statement:

SUPERCONDUCTING MATERIAL AND METHOD OF SYNTHESIS

Field of the Invention

5   The present invention relates to a superconducting compound and to a method of synthesising a superconducting compound, and, particularly, but not exclusively, a superconducting compound based on magnesium diboride and a method of synthesis thereof.

10

Background of the Invention

        Significant progress has been made in improving the critical current density (J_c) in various forms of MgB_2
15   since superconductivity in this compound was discovered [1]. This is largely attributable to the fact that grain boundaries in MgB_2 do not act as weak links, at least in low fields [2-4]. High J_c at a level of 10^5 A/cm^2 to 10^6 A/cm^2 at 20 K to 30 K for MgB_2 wires have been reported by
20   several groups [5-11]. However, J_c drops rapidly with increasing magnetic field. In all the studies on wires and bulk, J_c decreased more than 90% of its zero field value at 3 T within this temperature range due to the poor pinning ability of this material. If MgB_2 is to be useful in high
25   fields the flux pinning strength must be improved. Attempts to enhance flux pinning have resulted in an encouraging improvement in irreversibility fields (H_{irr}) and $J_c(H)$ performance in high fields by oxygen alloying in MgB_2 thin films [12] and by proton irradiation of MgB_2
30   powder [13]. The question is whether one can introduce effective pinning centres into MgB_2 bulk or wires by a normal process such as chemical doping.
        Several attempts have been made to improve flux pinning using chemical doping, but the results remain
35   controversial. Furthermore, the results for doping into MgB_2 are largely limited to addition rather than substitution. Zhao et al, and Feng et al have doped MgB_2

with Ti and Zr, showing improvement of J_c in self field [14,15]. However, there is evidence for improved pinning as the J_c drops off rapidly with increasing field (H_{irr} = 4T at 20K). Recently, Wang et al doped MgB_2 using nanoparticle

5   [16]. The results showed an improvement of irreversibility field (H_{irr}) at 4.2K for the doped sample. However, H_{irr} for the doped samples is not as good as the undoped ones at 20K. Cimerle et al., found that doping with a small amount of Li, Al and Si showed some increase in J_c, but there is

10   no improvement in H_{irr} [17]. By using high pressure synthesis, Prokhna et al showed an improvement of J_c-H behaviour for the Ta doped MgB_2 [18].

Summary of the Invention

15

In accordance with a first aspect, the present invention provides a superconducting material of formula $MgB_xSi_yC_z$ where X = 0 to 2, Y = 0 to 1 and Z = 0 to 1.

Preferably, X = 1 to 2, Y = 0.05 to 0.5 and Z = 0.05

20   to 0.5.

More preferably, X = 1.2 to 1.8, Y = 0.1 to 0.3 and Z = 0.1 to 0.3.

Advantageously, the factors of critical current density , irreversibility field and flux pinning

25   properties of MgB_2 are significantly improved by chemical doping with SiC, potentially paving the way for MgB2 to replace the current market leaders NbTi and Ag/bi2223.

superconducting material may be made into superconducting bulk, wires, thin films and various

30   articles and devices for any superconducting application.

In accordance with a second aspect, the present invention provides a superconductor incorporating the superconducting material of the first aspect of the invention.

35   In accordance with a third aspect the present invention provides a method of synthesising the superconducting material of the first aspect of the

present invention, comprising the steps of utilising starting materials Mg, B, Si and C. Preferably, these are in powder form. Preferably, the powders consist of nanoparticles.

5   In accordance with a fourth aspect, the present invention provides a method of synthesising the superconducting material of the first aspect of the invention comprising the steps of utilising starting materials Mg, B and SiC. Preferably, these are in powder

10 form. Preferably, the powders consist of nanoparticles.

      In accordance with a fifth aspect, the present invention provides a method of synthesising a superconducting material in accordance with the first aspect of the invention comprising the steps of utilising

15 starting materials MgB_2 and SiC or Si and C. Preferably, these are in powder form. Preferably the powders consist of nanoparticles.

      More broadly, in accordance with a sixth aspect, the present invention provides a method of producing a

20 superconducting material, comprising the steps of adding silicon carbide to a superconducting material.

      Preferably, the silicon carbide is added to by a way of addition.

      Preferably, the silicon carbide is added to by a way

25 of substitution.

      The silicon carbide may be added both by substitution and addition to the superconducting material.

      In accordance with a seventh aspect, the present invention comprises a superconducting material including a

30 silicon carbide codopant.

Brief Description of the Drawings

      Features and advantages of the present invention will

35 become apparent from the following description of examples thereof, by way of example only, with reference to the accompanying figures, in which;

Fig. 1. Critical transition temperature (T_c) measured using magnetic susceptibility versus temperature for $MgB_2(SiC)_x$ where x =0, 0.055, 0.11, 0.22 and 0.33.

Fig. 2. X-ray diffraction patterns for the undoped and
5 SiC-doped samples. Note that the MgB_4, MgO and MgB_4O_7 peaks increased significantly with increasing SiC.

Fig. 3 lattice parameters a and c as a function of the SiC content x. Note axis decreases with increasing SiC dopant and did not reach situation at x=0.33. The total variation of
10 axis from x=0 to x=0.33 is 0.012A. In comparison, single element doping with C axis reached a plateau at C content of 7at% of B where the variation of axis is 0.016A. This indicates that co-doping of Si and C into MgB_2 substantially reduced the variation of axis due to the counterbalance effect of si and C. This also
15 explains why the T_c drops very slow with increasing SiC dopant.

Fig. 4. Effect of SiC doping on the J_c - H dependence at 5 K (a) and 10 K (b) for all the samples at doping level from x=0 to x=0.33.

Fig. 5. A comparison of $J_c(H)$ for SiC doped sample at
20 x=0.11 at 20K with those for Ti doped [14], Y_2O_3 doped [16], thin film with strong pinning [12] and Fe-sheathed tape [21], which represent the state-the-art performance of MgB_2 in various forms.

Fig. 6 TEM image showing the high density
25 dislocations within the grains (a) and EDX analysis map showing the incorporation of C and Si into the grains of MgB_2.

Description of Examples and Preferred Embodiments
30

The superconducting composition and the processes for synthesis of the materials of the present invention can significantly enhance J_c and flux pinning. The results demonstrate that the claimed formula can be used for
35 fabrication of superconductors for high-field applications, as chemical doping is a readily achievable

and economically viable route to introduce effective flux pinning.

MgB$_2$ pellet samples were prepared by a reaction in-situ method which has been previously described in details [18]. Powders of magnesium (99%) and amorphous boron (99%) were well mixed with SiC additive at with the weight ratio of (Mg+2B)(SiC)$_x$ where x = 0, 0.055, 0.11, 0.22 and 0.33 for samples 1 to 5 respectively (Table 1). The mixed powders were loaded into Fe tubes. The composite tubes were groove-rolled, sealed in a Fe tube and then directly heated at preset temperatures to 950°C, for 3 hours in flowing high purity Ar. This was followed by quenching to liquid nitrogen. Table 1 gives the samples' parameters and selected results of T$_c$ and H$_{irr}$.

The magnetization of samples was measured over a temperature range of 5 to 30 K using Magnetic Property Measurement System (MPMS) and a Physical Property Measurement System (PPMS, Quantum Design) in a time-varying magnetic field of sweep rate 50 Oe/s and amplitude 5T and 9T, respectively. A magnetic J$_c$ can be derived.

Fig. 1 shows the transition temperature (T$_c$) and transition width ΔT$_c$ for the doped and undoped samples determined by ac susceptibility measurements. The T$_c$ onset for the undoped sample (38.6 K) is the same as reported by a number of groups. For the doped samples, the T$_c$ decreases with increasing doping level. It is striking to note that despite the large amount of non-superconducting phases present, the T$_c$ only drops 2.6K at the SiC doping level of x=0.33 (33at%of B). In contrast, the T$_c$ was depressed for almost 10K by 7% C substitution for B in MgB$_2$ [19] and 0.5at% Si substitution reduced T$_c$ for about 0.5K [20] These results suggest that the higher tolerance of T$_c$ of MgB$_2$ to SiC doping is attributable to the co-doping of C and Si because the average size of C (0.077nm) and Si (0.117nm) is exactly the same as that of B (0.097nm). It is evident that the co-doping with SiC counterbalanced the negative effect on T$_c$ of the single element doping.

Fig. 2 shows XRD patterns for the SiC doped and undoped samples. The X-ray scans were recorded using $Cu_{K\alpha}=1.5418Å$, and indexed within the space group P6/mmm. For the in-phase reflection which occur in Fig. 1 between

5 $2\theta=33°$ and $2\theta=34°$ indexed as (100), the centroid of the peak clearly shifts to higher 2θ values with increasing x, while in the same time the centroid of the peak which occur between $2\theta=51°$ and $2\theta=52°$, and indexed as (002), the shift is marginal to higher 2θ values with increasing x.

10 The lattice parameters, a and c of the hexagonal AlB_2-type structure of MgB_2 were calculated using these peak shifts as shown in Fig. 2. The continuous decrease of a with increasing SiC doping level indicates that B was substituted by C and Si. C substitution for B reached

15 saturation at 7at% of B[19] while the co-doping of Si and C for B raised the saturation level. At x=0.055, the sample consists of major phase with MgB_2 structure and minority phases: MgB_4, MgBO and MgO. The appearance of MgB_4 may be attributable to the substitution of SiC for B

20 position, resulting in excess of B. Some extra B was incorporated into MgO to form MgBO. It is also noted that all the three nonsuperconducting phases increased with increasing SiC dopant. At x=0.22, the amount of nonsuperconducting phases exceeded MgB_2. The extra oxygen

25 may be brought in by the SiC dopant which absorbed moisture or oxygen during storage. There are no SiC peaks indexed even up to x=0.33.

Fig. 5 shows the J_c(H) curves for doped and undoped samples at 5 K (a), 10 K (b). These results show the

30 following striking features. The J_c(H) curves for undoped samples shows a crossover with those for all the doped sample at higher fields. Although SiC doping at x greater than 0.22 caused a reduction of J_c in low fields, it is important to note that the J_c for all the doped samples

35 drops with increasing field much more slowly than for the undoped one. In particular, J_c curves for the doped samples x=0.22 and 0.33 show an exponential relationship with

magnetic field up to the measurement field limit, while the J_c curve for the undoped sample shows a rapid bending downward. The J_c for the doped sample at x=0.11 reached $1.3 \times 10^5 A/cm^2$ at 4T and increased more than 30 times that of the undoped sample at 6T and 5K. It is also interesting to note is that at 5K and 10K $J_c(H)$ for higher doping level samples (4 and 5) declined faster than those of low doping samples (2 and 3). But, at 20K all doped samples showed nearly parallel $J_c(H)$ curves while at 30K, x=0.33 doped sample showed even slower J_c drop with field than all others, indicating that a stronger pinning enhancement effect at high temperatures than at low temperatures.

Fig. 4 shows a comparison of J_c- H behaviour for x=0.11 SiC doped sample with data reported from literature at 20K. It is evident that despite the low density and unoptimised composition, the J_c for the SiC doped sample drops slower than other element doped samples [14-16], the best Fe/MgB_2 tape [17] and even close to the thin film with strong pinning [12]. At 20K, the sample with x=0.11 has a J_c value of $18,000A/cm^2$ at 4T, 100 times the control sample which was made at the same batch, 8 times that of the state-the-art Fe/MgB_2 tape [17] and comparable to the Ta doped MgB_2 made using high pressure synthesis (2GP) [21]. These are the best J_c values ever reported for bulk and wires made under normal conditions. The H_{irr} for x=0.11 doped sample is 6T at 20K and 8T at 10K, compared to 4T and 6T of the undoped one, respectively.

Regarding the mechanism of the enhancement of J_c at higher fields, it is necessary to recognize the special features of SiC doping. First, in contrast to previous work on doping for improving J_c [14-16, 20], SiC doping has no densification effect as evidenced by the fact that the density of doped samples is $1.2G/cm^2$, independent of doping level. This is understandable because SiC has very high melting point and would not act as sintering aid at the temperature range of 800°C to 950°C. Second, SiC doping takes place in the form of substitution and/or addition

while in the previous work [14-16, 20] the element doping is in the form of additives, not incorporating into lattice. Doping MgB_2 with Ti and Zr showed an improvement of J_c in self field and 4K [14,15]. However, there is

5 evidence for improved pinning as the J_c drops off rapidly with increasing field (H_{irr} = 4T at 20K). Doping MgB_2 using Y_2O_3 nanoparticles showed an improvement of irreversibility field (H_{irr}) at 4.2K, but H_{irr} for the doped samples is not as good as the undoped ones at 20K [16]. Cimerle et al.,

10 found that doping with a small amount of Li, Al and Si showed some increase in J_c, but there is no improvement in H_{irr} [19]. It is evident that the additive pinning is more effective at low temperatures while the additives at the grain boundaries decouple the grains at high temperatures.

15 There are two potential pinning mechanisms in the SiC doped samples: intrinsic pinning due to substitution and impurity pinning or a combination of these two. The high fraction of substitution by both Si and C could result in lattice defects, which can act as effective pinning sites

20 which are intrinsic nature and independent of temperature. The high content of MgO and other impurity phases in the SiC doped samples could also be potential pinning centers, consistent with the results obtained from a thin film with strong pinning where the ratio of Mg;B:O reached

25 1.0:0.9:07 [12]. We have attempted to dope fine particle MgO into MgB_2. But the results did not show any improvement in J_c. This indicates that the way to introduce the impurities is very critical. When SiC react with liquid Mg and amorphous B at the sintering temperatures, the

30 nanoparticles may act as nucleation sites to form MgB_2 and other phases. Some nanoparticles can be included within the grains as inclusions. Thus, the reaction induced products are highly dispersed in the bulk matrix. These arguments are supported by study of microstructures. SEM

35 examination revealed that the grain size is smaller than 100nm. The EDX analysis results showed the Mg:Si ratio is identical across all the entire sample area, indicating

that the phase distribution is homogeneous. The results of
the present work suggest that a combination of
substitution and highly dispersed additives induced
through the substitution is responsible for the enhanced
5   flux pinning in SiC-doped MgB_2. The substitution induced
defects and order parameter fluctuation may play more
important role than the impurity pinning.

It should be pointed out that the density of the
samples is only about 1.2 g/cm^3. Thus, the J_c values for
10   both the doped and undoped samples are far from optimum.
We can anticipate that a higher J_c and better flux pinning
enhancement can be achieved if the density of the samples
is further improved.

From the study of effect of the purity of the
15   precursor materials [22], we noted that even 95% pure B
degraded the J_c appreciably. So it is necessary to use high
purity B (98% or above). The cost for B increases
significantly with increasing purity. The main cost for
making MgB_2 conductors will be the high purity B. Because C
20   and Si are abundant, inexpensive and readily available
materials if part of B can be replaced by co-doping of C
and Si or SiC the overall cost for making MgB_2 conductors
will be reduced. Furthermore, the SiC doping has already
shown the significant benefit to enhance flux pinning. It
25   is evident that the future MgB_2 conductors will be made
using a formula of $MgB_xSi_yC_z$ where $x+y+z \geq 2$, in stead of pure
MgB_2.

In summary, we have demonstrated that the critical
current density, irreversibility field and flux pinning
30   properties of MgB_2 in bulk form can be significantly
improved by a readily achievable and economically viable
chemical doping with SiC, paving the way for MgB_2 to
potentially replace the current market leader, Nb-Ti.

35

Table 1

Sample No	SiC wt%	At. Ratio MgB$_2$:SiC	Density G/cm^2	$T_{c(0)}$, K	H_{irr}(T) 10 K	H_{irr},T 20 K	H_{irr}(T) 25 K	H_{irr},T 30K
1	0	1 : 0	1.20	38.6	6.2	4.0	3.6	2.2
2	5	1 : 0.055	1.21	37.8	7.2	5.1	4.0	2.5
3	10	1 : 0.11	1.22	36.8	8.0	5.8	4.5	2.5
4	20	1 : 0.22	1.17	36.6	5.5	3.7	3.5	1.8
5	30	1 : 0.33	1.30	36	5.2	4.0	3.2	2.5

Example 1: Synthesis of $(Mg+2B)_{1-x}(SiC)_x$ through
5   nanoparticle SiC doping into MgB$_2$

$(Mg+2B)_{1-x}(SiC)_x$ samples were prepared by a reaction
in-situ method according to the stepsin cliam 6. Powders
of magnesium (99%) and amorphous boron (99%) were well
10   mixed with SiC additive with the atomic ratio of $(Mg+2B)_{1-x}(SiC)_x$ where x = 0, 0.057, 0.117, 0.23 and 0.34 for
samples 1 to 5 respectively (Table 1). The mixed powders
were loaded into Fe tubes. The composite tubes were
groove-rolled, sealed in a Fe tube and then directly
15   heated at preset temperatures to 950°C, for 3 hours in
flowing high purity Ar. This was followed by quenching to
liquid nitrogen temperature. Table 1 gives the important
information on the present three samples, with 1, 2, and 3
being the undoped and doped with 5wt% and 10wt% MgO
20   respectively.
        The magnetization of 1.0 x 1.0 x 0.8 mm^3 samples was
measured over a temperature range of 5 to 30 K using a
Physical Property Measurement System (PPMS, Quantum
Design) in a time-varying magnetic field' of sweep rate 50
25   Oe/s and amplitude 9T. A magnetic J_c can be derived from
the height of the magnetization loop ΔM using a suitable
variant of the "semi-Bean" relationship $\Delta M = k\, J_c d$, where k
is a constant and d the thickness that the sample presents
to the applied field. Based on the full sample size, the
30   magnetic J_c is calculated using the relationship for a
plate in a perpendicular field: $J_c = 20\Delta M/(a-a^2/3b)$. J_c

versus magnetic field up to 9 T for the samples at 5 K, 10 K, 20 K, and 30 K has been measured. Because of flux jumping the low field J_c below 10 K cannot be measured.

5   Example 2: Synthesis of $MgB_{2-x}(SiC)_x$ using nanoparticle SiC to react with Mg and B

      $MgB_{2-x}(SiC)_x$ samples were prepared by a reaction in-situ method comprising the steps described in cliam 6.
10   Powders of magnesium (99%) and amorphous boron (99%) were well mixed with SiC additive with the ratio of $MgB_{2-x}(SiC)_x$ where X = 0, 0.02, 0.05, 0.1, 0.15, 0.2, 0.5, 1.0, 1.5 and 2.0. The mixed powders were loaded into Fe tubes. The composite tubes were groove-rolled, sealed in a Fe tube
15   and then directly heated at preset temperatures to 900°C, for 3 hours in flowing high purity Ar. This was followed by quenching to liquid nitrogen temperature.

Example 3: Synthesis of $MgB_{2-x-y}Si_xC_y$ using Mg, B, Si and C
20   powders

      MgB_2 pellet samples were prepared by a reaction in-situ method comprising the steps described in claim 6. Powders of magnesium (99%) and amorphous boron (99%) were
25   well mixed with SiC additive with the ratio of $Mg+B_{2-x}(SiC)_x$ where x = 0, 0.02, 0.05, 0.1, 0.15, 0.2, 0.5, 1.0, 1.5 and 2.0. The mixed powders were loaded into Fe tubes. The composite tubes were groove-rolled, sealed in a Fe tube and then directly heated at preset temperatures to 900°C,
30   for 3 hours in flowing high purity Ar. This was followed by quenching to liquid nitrogen temperature.
      Although the above embodiments specifically relates to magnesium diboride superconducting material it is possible that silicon carbide codopant could be useful for
35   other superconducting materials.
      A suitable process for utilising the superconducting material of the present invention would be as follows:

- Mixing powders of precursor materials as discussed above.
- Ball milling the mixture to achieve homogeneity.
- Loading the mixture in to metal or alloy dies, containers, tubes for forming and shaping, the metals including iron, stainless steel, nickel and various alloys.
- Making the pmixtures or the composites in to bulk, wires, tapes and various shaped articles and devices through mechanical deformation including pressing, drawing, rolling, swaging and casting etc.
- Sintering the composites at temperatures of 650^0C to 1000^0C for 10 minutes to 10 hours, preferably at 800 to 950^0C for 10 minutes to 3hours.

Modifications and variations as would be apparent to a skilled addressee are deemed to be within the scope of the present invention.

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CLAIMS

1. A superconducting material of formula $MgB_xSi_yC_z$ where $X = 0$ to 2, $Y = 0$ to 1 and $Z = 0$ to 1.

5   2. A superconductor incorporating the superconducting material of claim 1.

3. A method of synthesising the superconducting material of claim 1 comprising the steps of utilising starting materials Mg, B, Si and C.

10   4. A method of synthesising the superconducting material of claim 1 comprising the steps of utilising starting materials Mg, B and SiC.

5. A method of synthesising a superconducting material in accordance with claim 1 comprising the steps of

15   utilising starting materials MgB_2 and SiC or Si and C.

6. A method of producing a superconducting material, comprising the steps of adding silicon carbide to a superconducting material.

20   7. A superconducting material including a silicon carbide codopant.
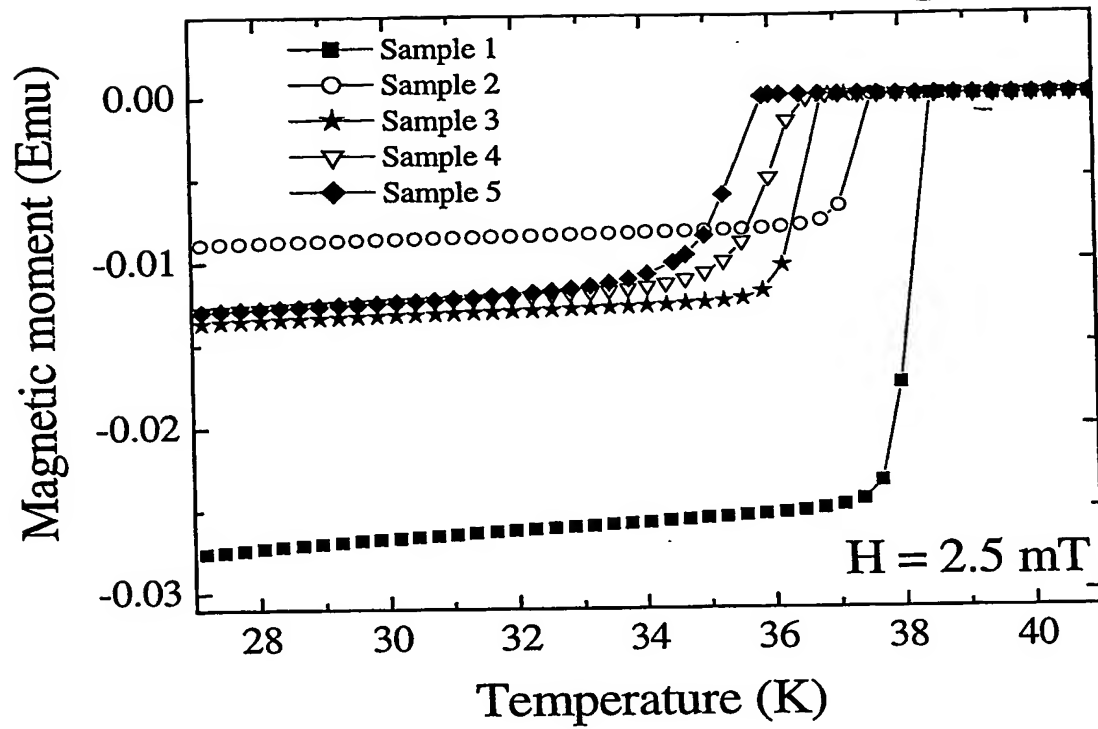
Fig. 1. Critical transition temperature (T_c) measured using magnetic susceptibility versus temperature for $MgB_2(SiC)_x$ where x =0, 0.057, 0.115, 0.23 and 0.34.
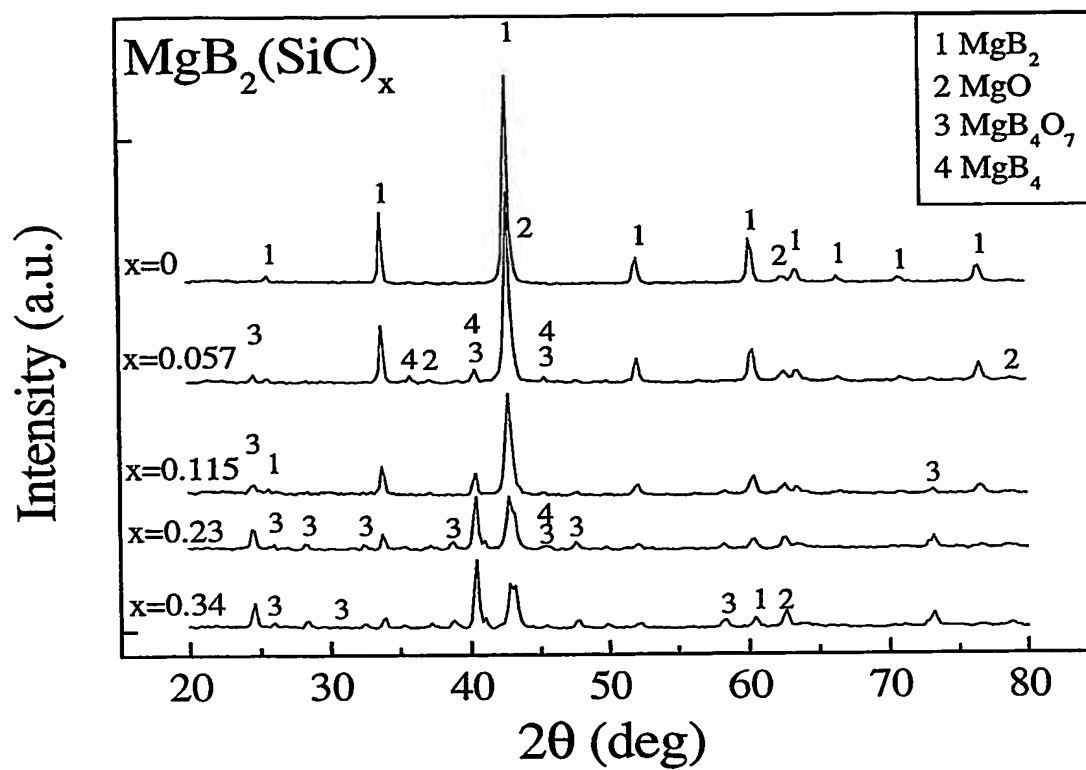
Fig. 2. X-ray diffraction patterns for the undoped and SiC-doped samples. Note that the MgB_4, MgO and MgB_4O_7 peaks increased significantly with increasing SiC.
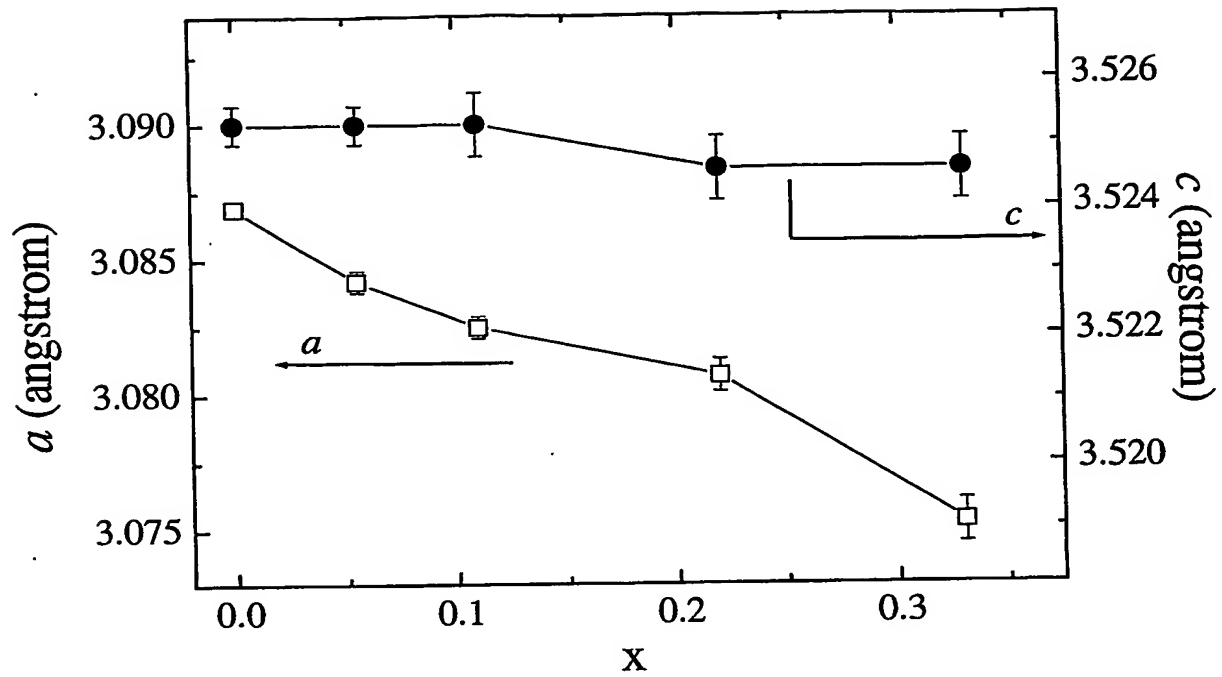
Fig. 3 lattice parameters a and c as a function of the SiC content x. Note a decreases with increasing SiC dopant and did not reach situation at x=0.34. The total variation of a from x=0 to x=0.34 is 0.012A. In comparison, single element doping with C a reached a plateau at C content of 7at% of B, where the variation of a was 0.016A [17]. This indicates that co-doping of Si and C into MgB_2 substantially reduced the variation of a due to the counter-balance effect of Si and C. This also explains why the T_c drops very slow with increasing SiC dopant.
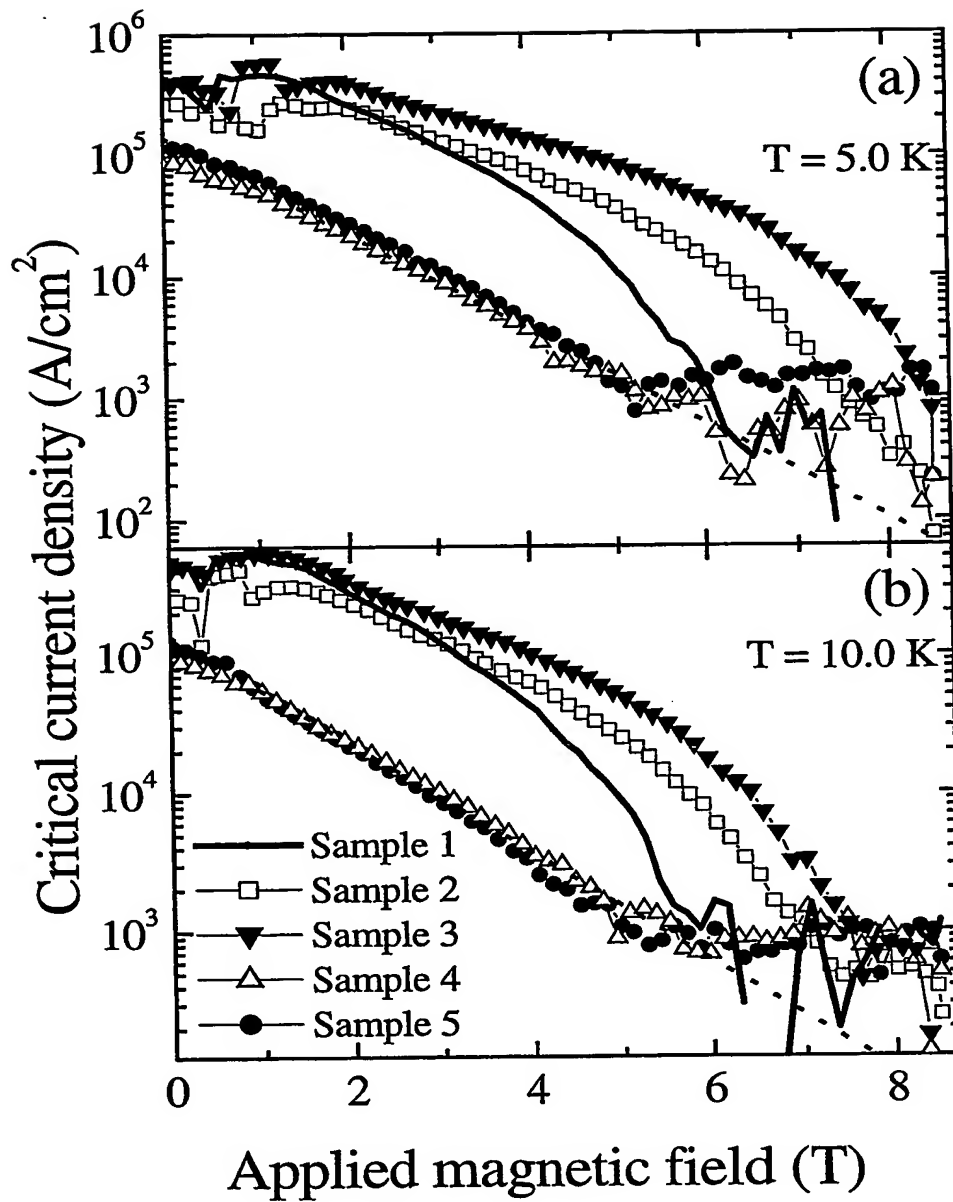
Fig. 4. Effect of SiC doping on the $J_c - H$ dependence at 5 K (a) and 10 K (b) for all the samples at doping level from x=0 to x=0.334.
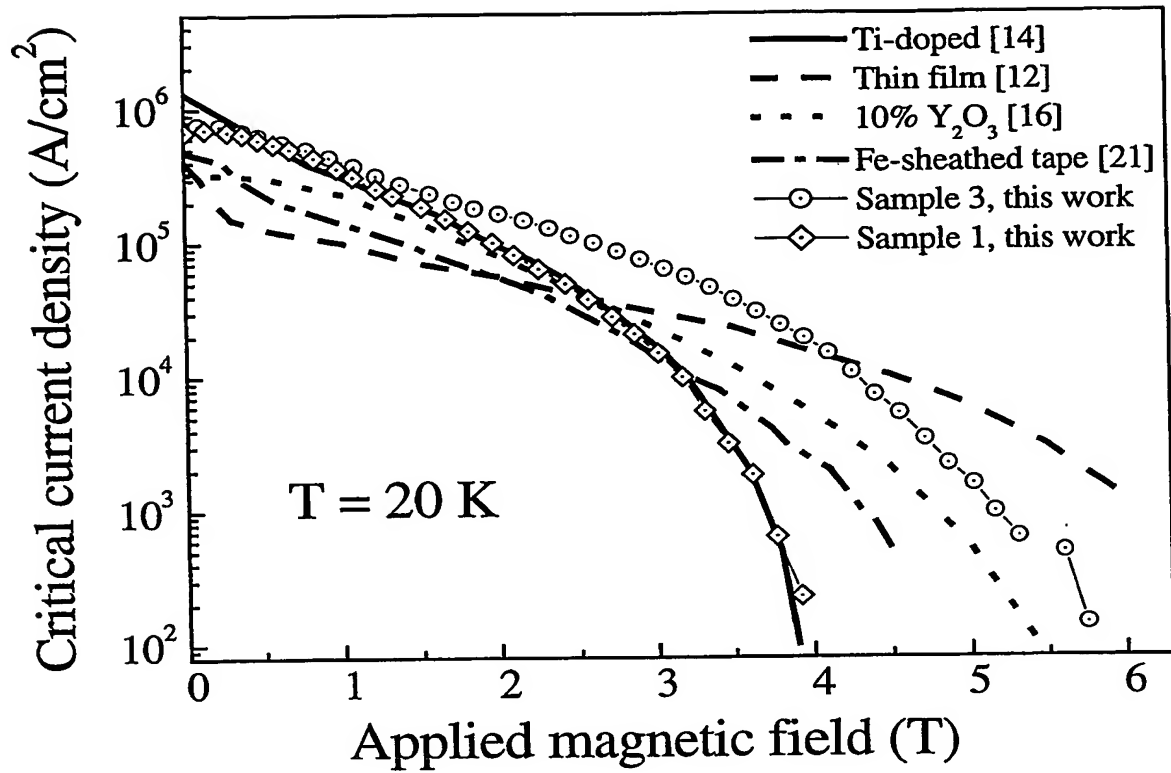
Fig. 5. A comparison of $J_c(H)$ for SiC doped sample at x=0.115 at 20K with those for Ti doped [14], Y_2O_3 doped [16], thin film with strong pinning [12] and Fe-sheathed tape [21], which represent the state-of-the-art performance of MgB_2 in various forms.
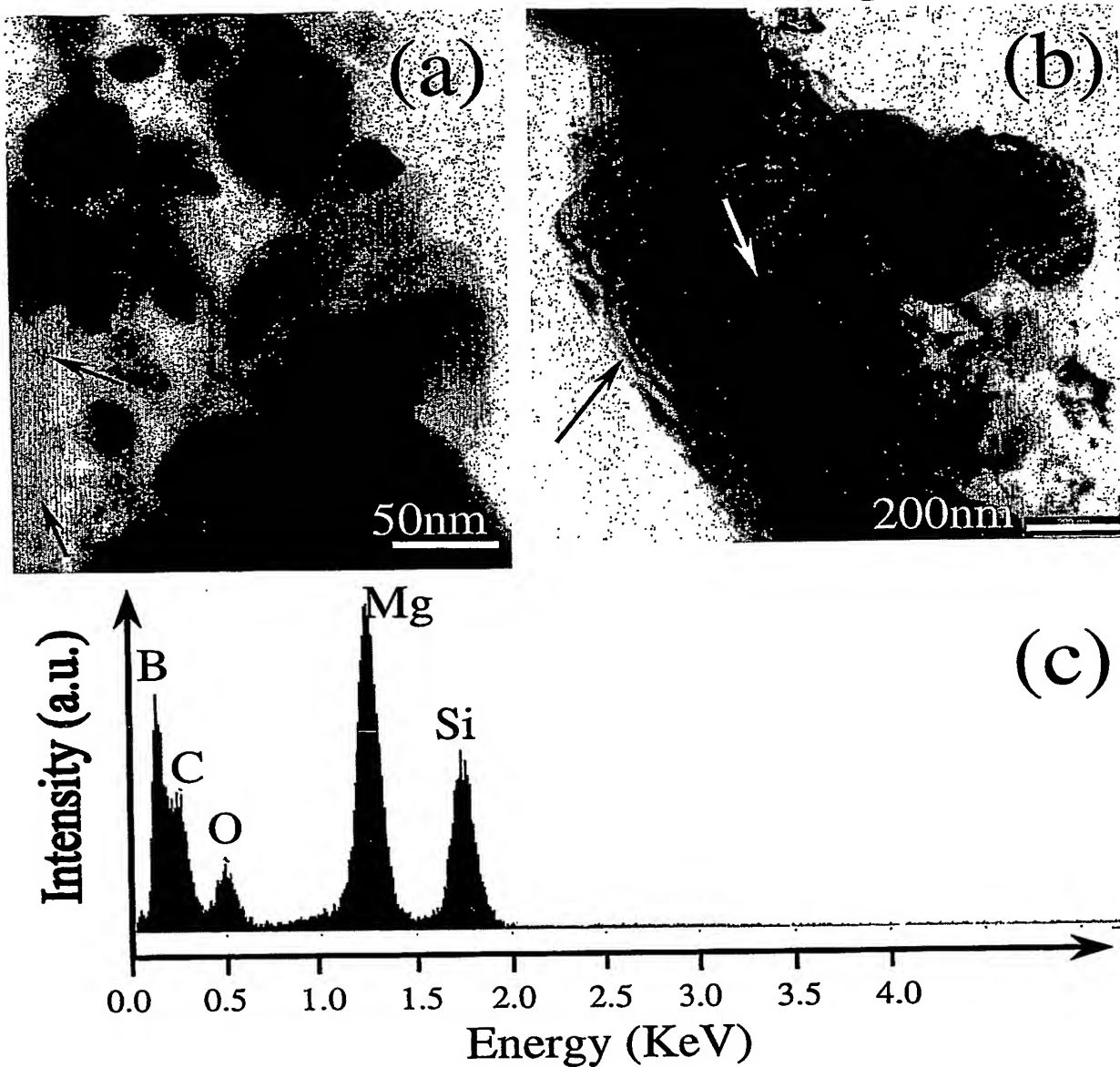
Fig. 6. TEM images (a,b) and EDX analysis (c) of the doped sample 3 (a,b). 20-500 nm large MgB$_2$ grains are shown in both TEM images. Impurities particles are < 10 nm (shown by arrows in (a)). A large number of dislocations (shown by arrows in (b)) is present in the MgB$_2$ grains. The EDX analysis for one of these grains shows that both Si and C are present within the grain (c).